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Fifteenth Progress Report
Covering the Period January 25, 1967 to May 15, 1967

Slush Hydrogen Fluid Characterization
and Instrumentation Analysis

Prepared for

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1. PERSONNEL

During the period January 25, 1967 to May 15, 1967, the following personnel worked on this project:

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2. ACCOMPLISHMENTS DURING THE PERIOD

Progress during the period has been in three general areas: the hydrogen flow system, solid hydrogen particle study, and the N.R. A. quality determination study.

2.1 Slush Hydrogen Flow System

Pump Calibration with Nitrogen

Slush solid fraction determination tests were made with nitrogen by measuring the gas pumped off while making slush by the freeze-thaw method. A check on the computed solid fraction of the slush generated was made by melting the slush with heaters in the bottom of the generator. The agreement was poor. It was decided that additional points for the curve of inlet pressure versus pumping rate should be taken to check the pump calibration scatter. Many additional points were taken by pumping known volumes of gas from a gas holder. A curve indicating the scatter is shown in figure 1.

Pump Calibration with Hydrogen

A similar pump calibration curve was generated for hydrogen gas as shown in figure 2. There is considerable scatter near and below the

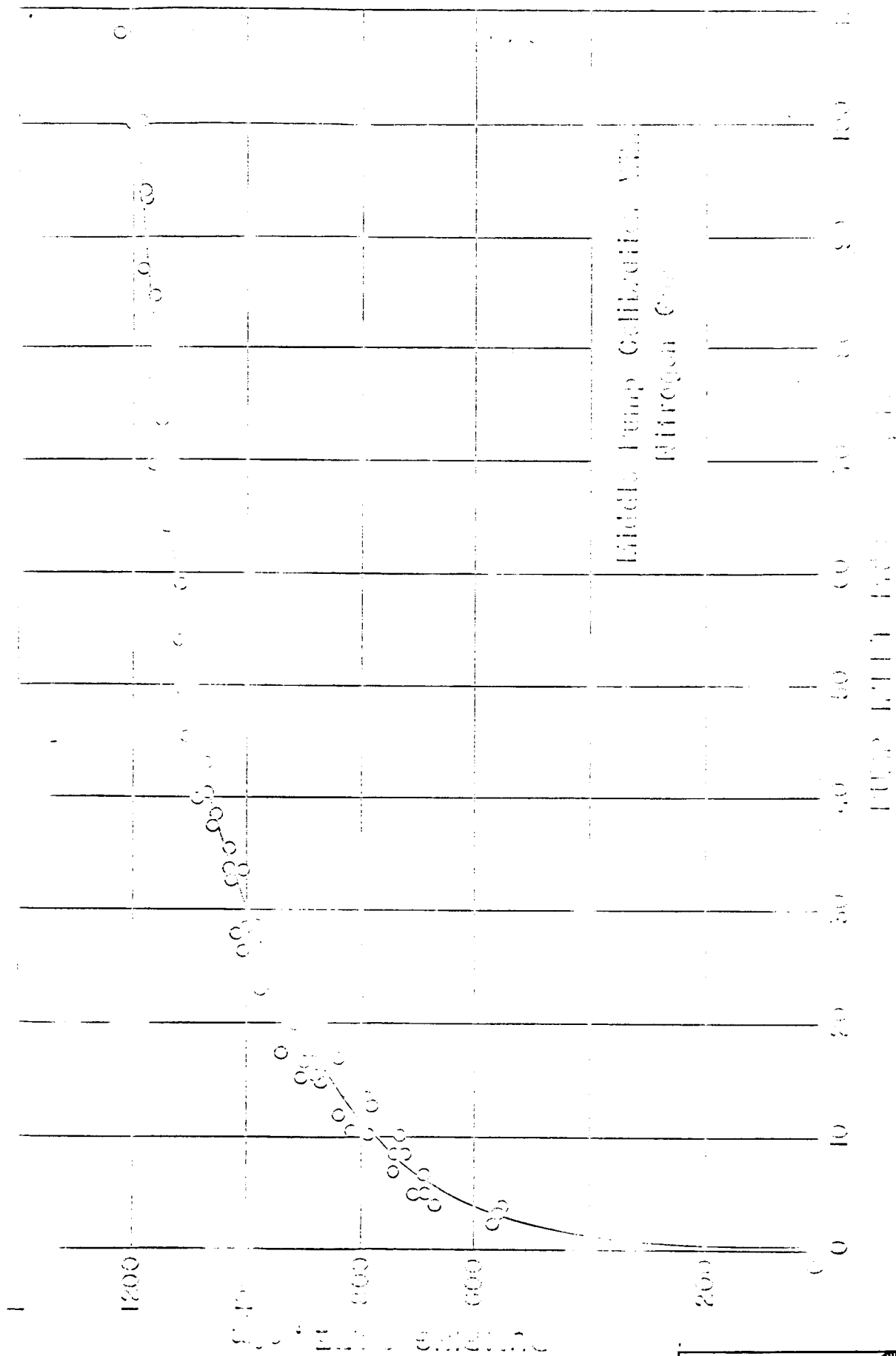


Figure 1.



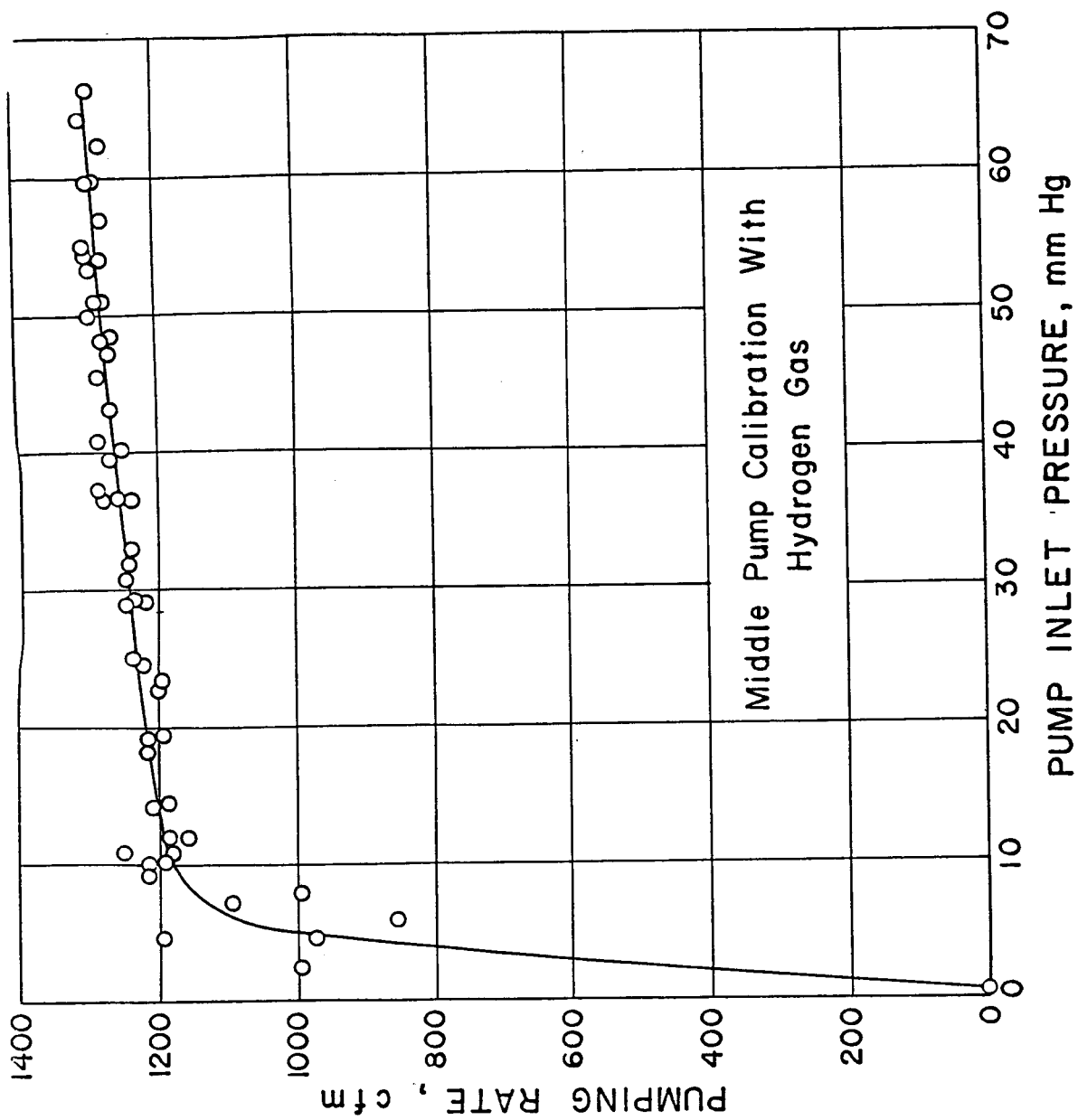


Figure 2

knee of the curve. The extent of the scatter above and below the curve is shown in figure 3. A similar plot in terms of grams per minute is shown in figure 4, which indicates that the maximum scatter is about ± 3 grams/minute. Comparing this figure to an average pump run in which 1334 gms were pumped in 800 seconds, the percent error can be calculated. The average pumping rate for the pump run was 202 gms/minute. The scatter of 3 grams/minute gives an error of ± 1.5 percent. During the freeze-thaw cycle the pump operates approximately half the time near the triple-point pressure of 52 torr and the other half at zero flow condition of 2 torr. Operation time between those pressures is a short duration transient. The curve in figure 2 is the calibration presently being used to determine the amount of gas pumped off during a pump run.

Freeze-Thaw Production Parameters

The minimum pumping rate necessary to generate slush by the freeze-thaw method was determined. The pump used has a rate of ≈ 1270 CFM @ 52 torr and is more than adequate for generating slush in a 30 inch diameter dewar. For this experiment, the maximum opening of the butterfly valve, between the generator and vacuum pump, was varied. Slush was then made by the freeze-thaw method in the normal manner with limited butterfly valve opening. The freeze-thaw generation was visually observed in the generator and the mass pumped off was computed as in a normal pump run.

No apparent difference in the manner of slush generation was observed until the maximum opening of the butterfly valve was limited to ≈ 30 degrees. At this setting slush production became marginal. The lowest average pumping rate at which slush generation was not noticeably affected was $88 \text{ ft}^3/\text{min}/\text{ft}^2$ of liquid surface area. It must be pointed out that the cubic feet above are at 300 K and 52 torr pressure. Also, this is the average pumping rate. Since the valve is closed approximately half the period of a freeze-thaw cycle, it appears that a system with a pumping

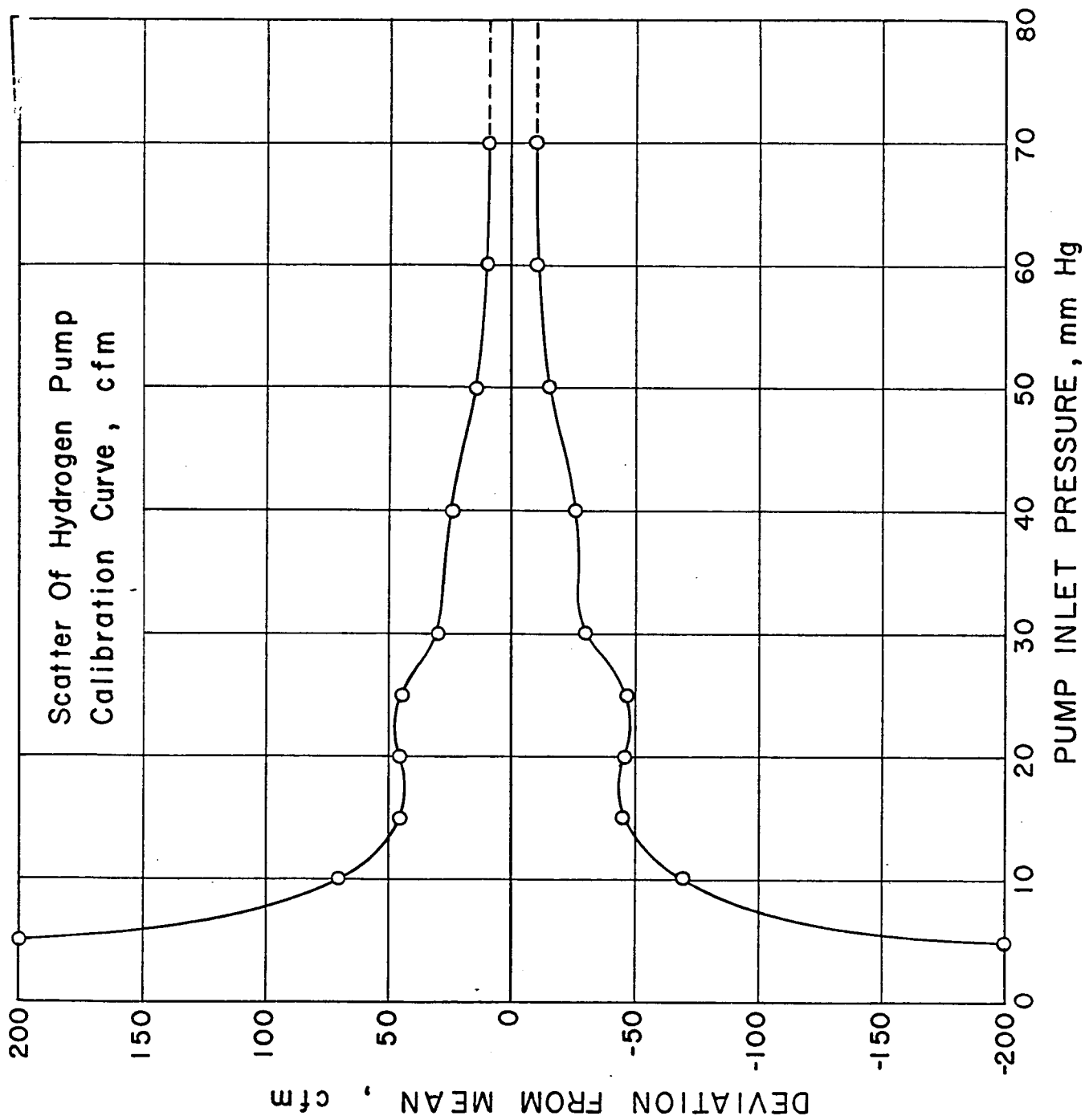


Figure 3

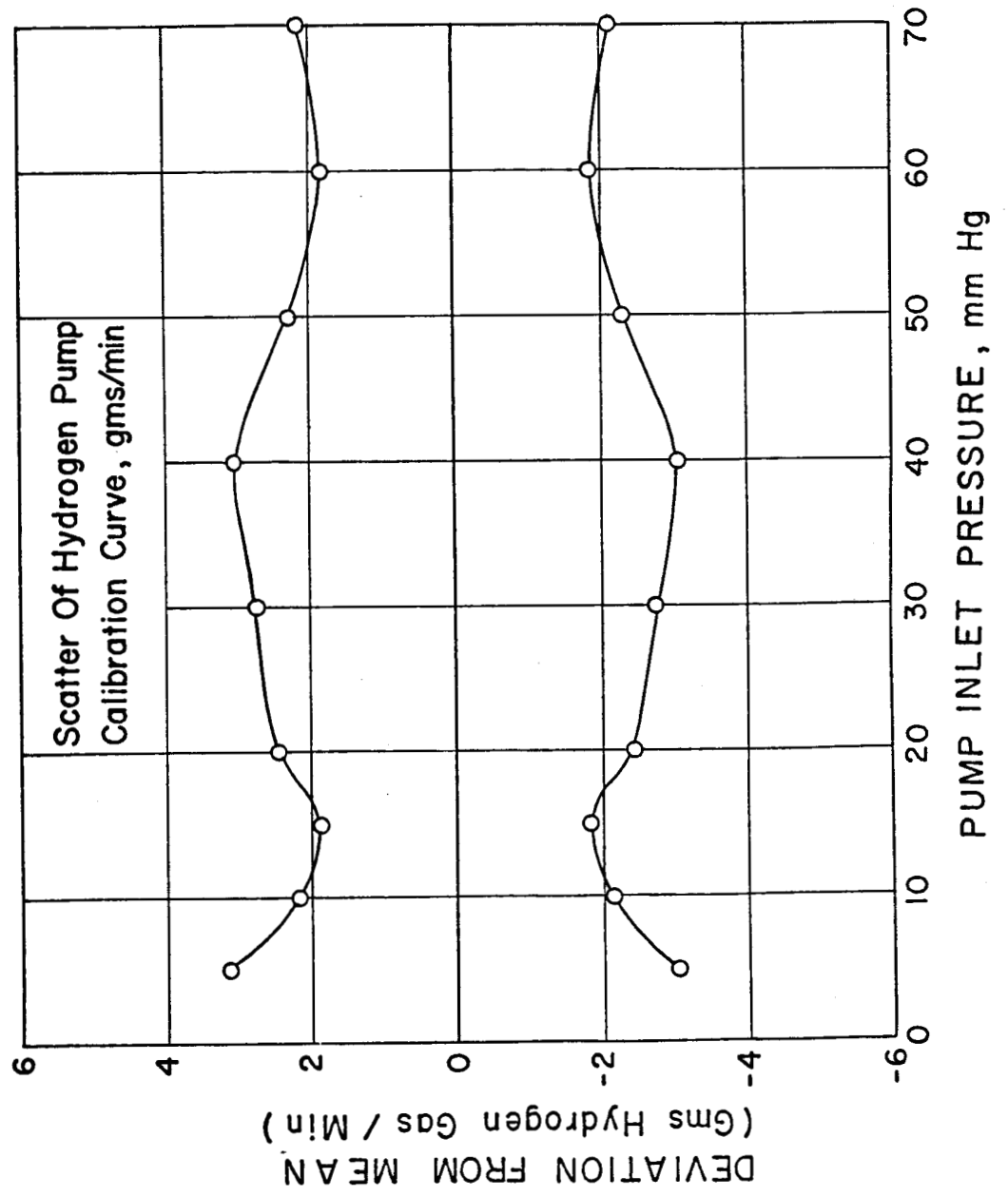


Figure 4

capacity of $2 \times 88 \text{ ft}^3/\text{min}/\text{ft}^2$ would probably be the minimum necessary to effectively and continuously generate slush by the freeze-thaw method. With a ballast tank in the pumping system, the pumping rate could be reduced to the $88 \text{ ft}^3/\text{min}/\text{ft}^2$.

Installation of Pressure Regulators

During the initial flow runs, the ullage pressure was controlled manually with a hand valve. The flow data indicated that the ullage pressure was too erratic for precise flow measurements and that a more precise method of flow regulation was necessary. To correct this, two precision regulators were installed into the flow system. These allow precise regulation of positive pressure in the generator and receiver dewars.

With these regulators, the transfer line differential pressure varies approximately 10 torr during a flow run as compared to 30 torr variations before they were installed. During flow runs the receiving dewar is vented to barometric pressure through large feather valves in the vacuum pump.

Generator Calibration

Early flow data indicated that the generator volume calibration was possibly in error. The flow was being computed using the measured volumes between each set of liquid level sensors. Variations in flow were indicative of an error in the volume calibration between some of the resistors. A recheck of the volume calibration was felt necessary.

The generator volume calibration was rechecked twice, using liquid nitrogen and measuring the boil off gas. The variations in volume averaged 0.5 liters for each increment and up to 1.5 liters maximum. A volume for each increment was averaged from the three calibrations. The 0.5 liter average variation among the three calibrations indicates a volume calibration error of this magnitude between any two liquid level sensors.

At this point it was decided that the volume error was too large when computing flow between each set of sensors, and that flow should be computed by measuring the volume from sensor #5 to #1 or from #6 to #2. This would give an average uncertainty of 0.5 liters in 132 rather than 0.5 liters in 33. At present the flow is computed with a volume measurement from sensor #5 to #1, and this improves the flow data considerably.

Triple-point Liquid Flow Runs

The first flow loop runs for pressure drop data were made with triple-point liquid hydrogen. Triple-point liquid was used for several reasons. Its temperature is the same as slush; therefore, thermally induced pressure disturbances in transducers would be similar to slush. Also, the results of pressure drop data in the test section could be compared to pressure drop predicted by classical methods.

The procedure for the triple-point liquid runs was as follows. The generator was filled with liquid hydrogen. The liquid was reduced to triple-point temperature by pumping. Triple-point conditions were assured by making a very small quantity of solid and by thorough mixing. The generator was then pressurized with helium gas to the desired pressure level. The receiver was vented through feather valves to the atmosphere. The flow loop valves were opened. Pressure in the generator was maintained at the desired level by the regulators described previously. The liquid volume flow was determined with the liquid level sensors. Pressure data were taken at the points shown in figure 5. Liquid level was read every two-tenths second and pressure every second. Data were recorded directly on magnetic tape which is subsequently processed in the computer. Results of the triple-point liquid runs are shown on figure 6. All triple-point liquid runs are plotted and all except the point at a flow rate of 0.263 liters per second are within ± 2.6 percent of the pressure drop predicted by classical methods. At the flow rate of .263 liters per second the velocity in the line

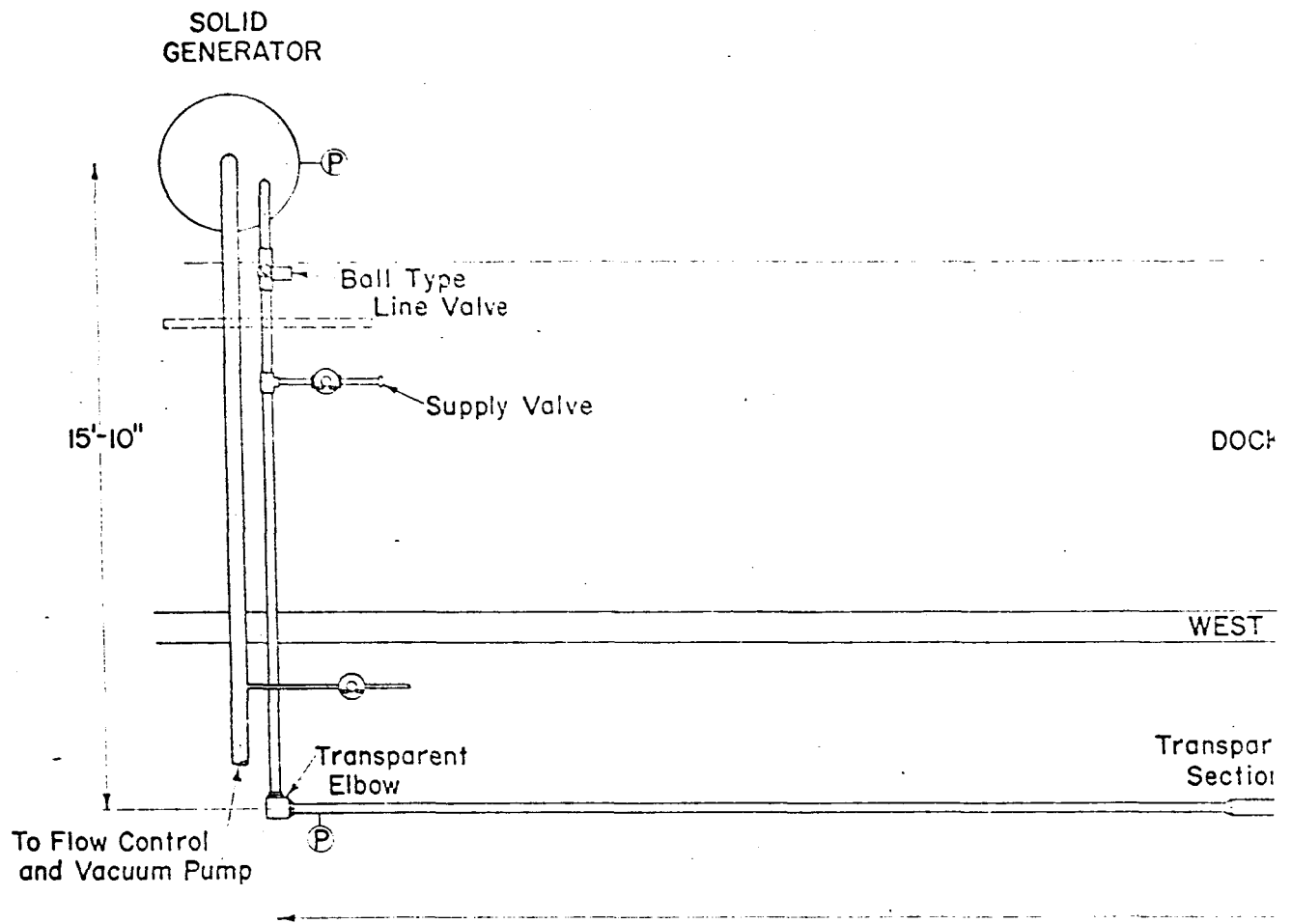
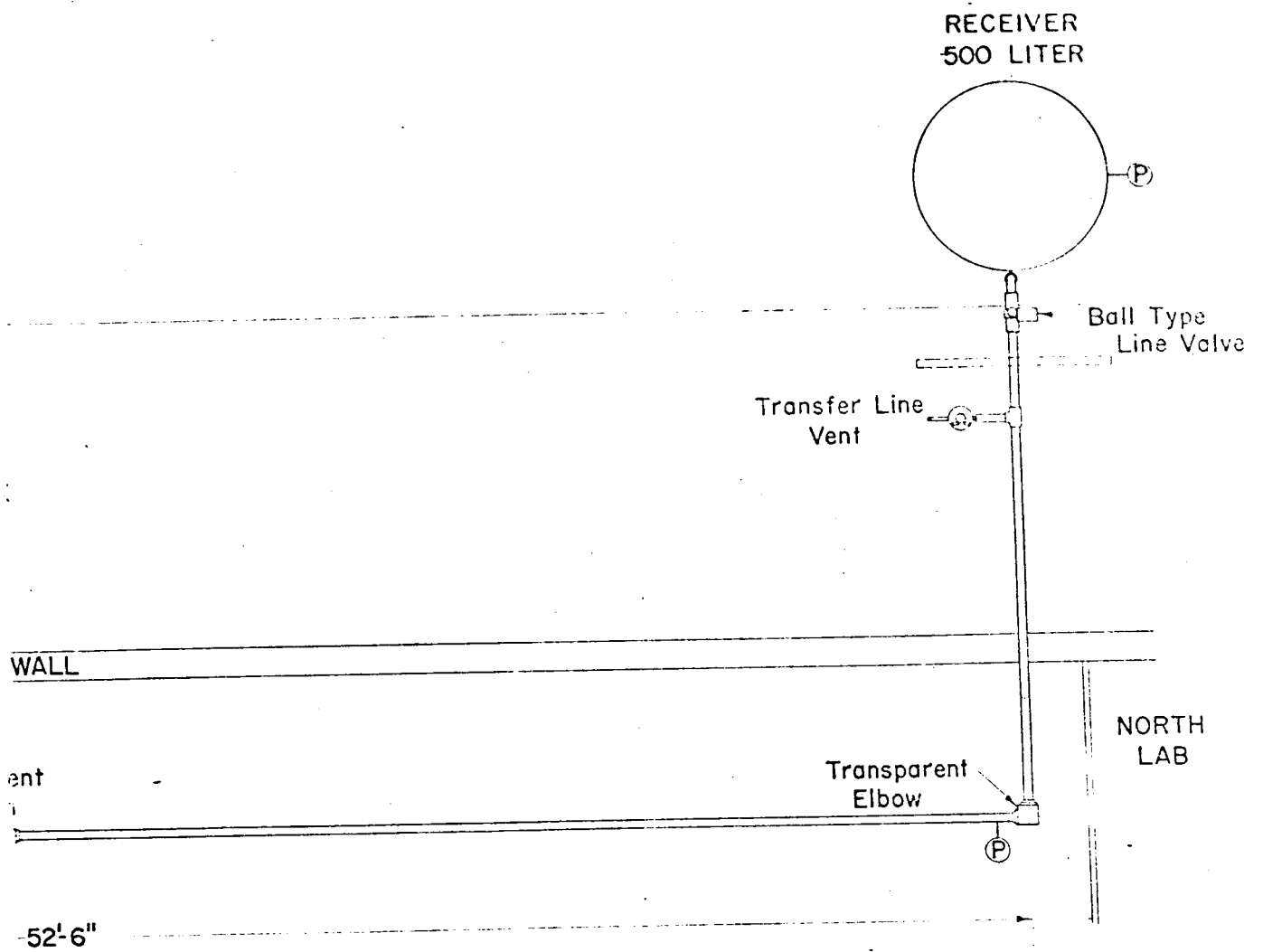


Figure 5. Flo



w Loop Schematic

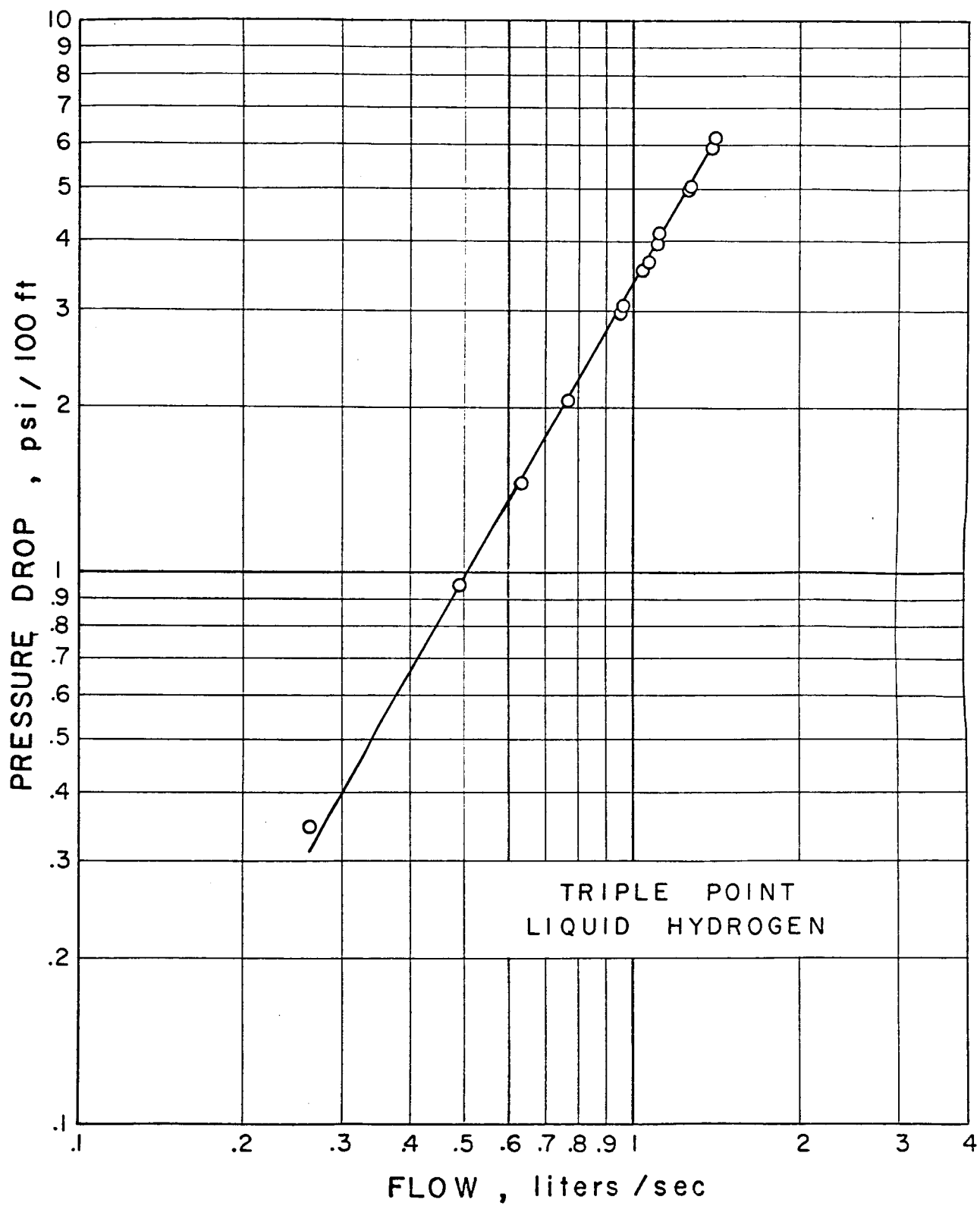


Figure 6

is 122 cm per second. The velocity at the highest flow shown is approximately 645 cm per second. For comparison purposes the data have been converted to a 100 feet equivalent length. More data at the lower flow rates will be taken to establish correlation between triple-point liquid and slush flow.

Slush Solid Fraction Determination

Twenty-seven runs were made with slush varying in solid fraction from 0.29 to 0.43. The slush solid fraction for the flow runs was determined by the mass-energy balance method. This method is described in detail by Daney and Mann [1966]. The liquid was reduced to triple-point temperature by the same method used for the triple-point liquid runs. The mass and energy inventory is maintained from the triple-point liquid conditions. The generator was filled to a given liquid level sensor with triple-point liquid establishing the initial mass. The data acquisition system was then turned on and pumping was continued, making slush by the freeze-thaw process. The data taken during the slush formation were the liquid level and the pump suction pressure. Both parameters are recorded every two-tenths seconds. These data are then reduced by the computer. From the pump calibration curve shown in figure 2, the mass of gas pumped off is determined. Using the initial mass, the mass pumped off, and the heat leak of the dewar, the slush solid fraction is calculated.

Slush Flow Runs

The results of the twenty-seven runs made with slush are shown in figure 7. The line shown on the figure is for triple-point liquid. The slush solid fraction of the points is as noted.

The procedure for slush flow runs was essentially the same as for the triple-point liquid runs with the exception that continuous stirring was required to maintain homogeneity during the flow. In the initial runs made with

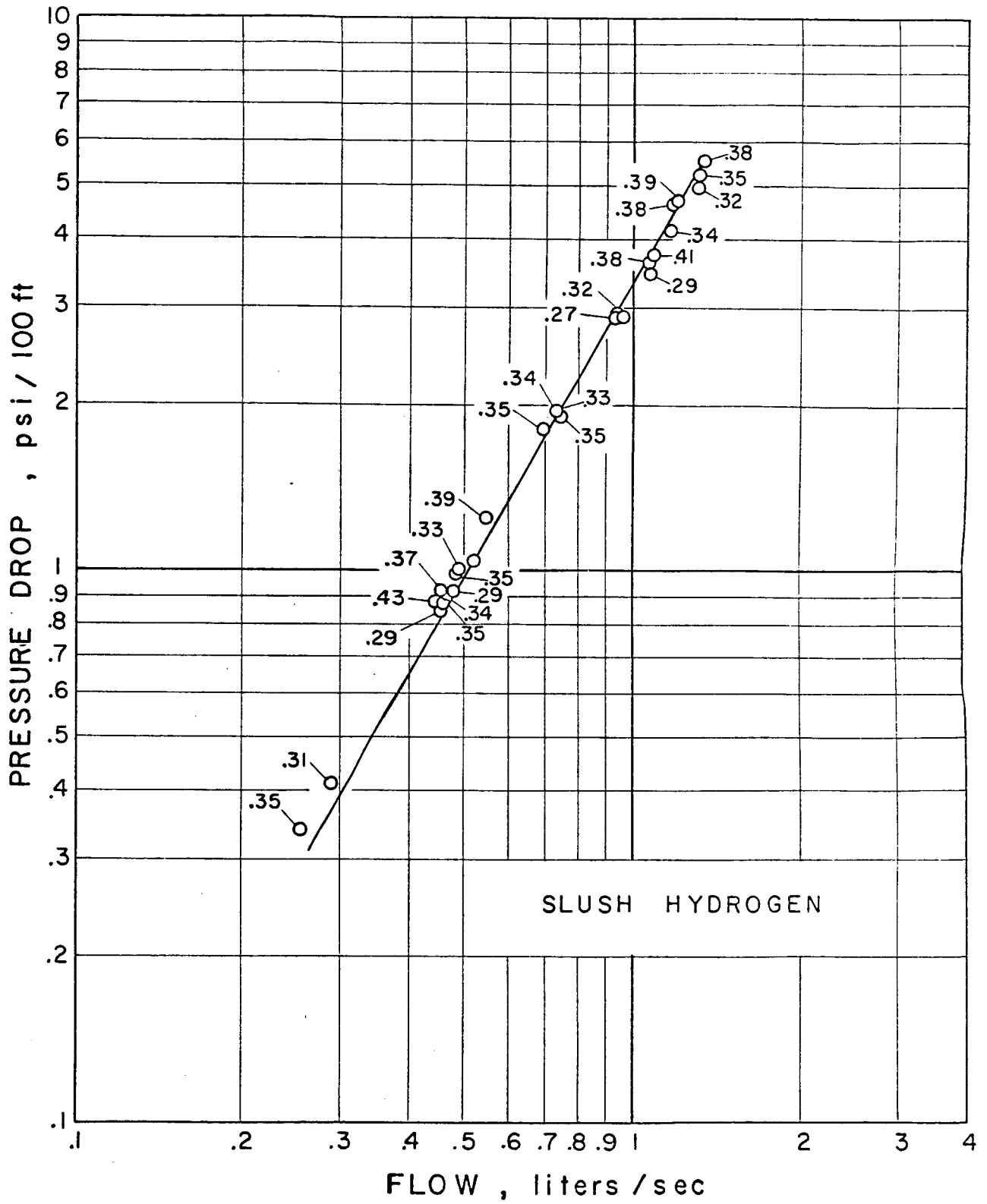


Figure 7.

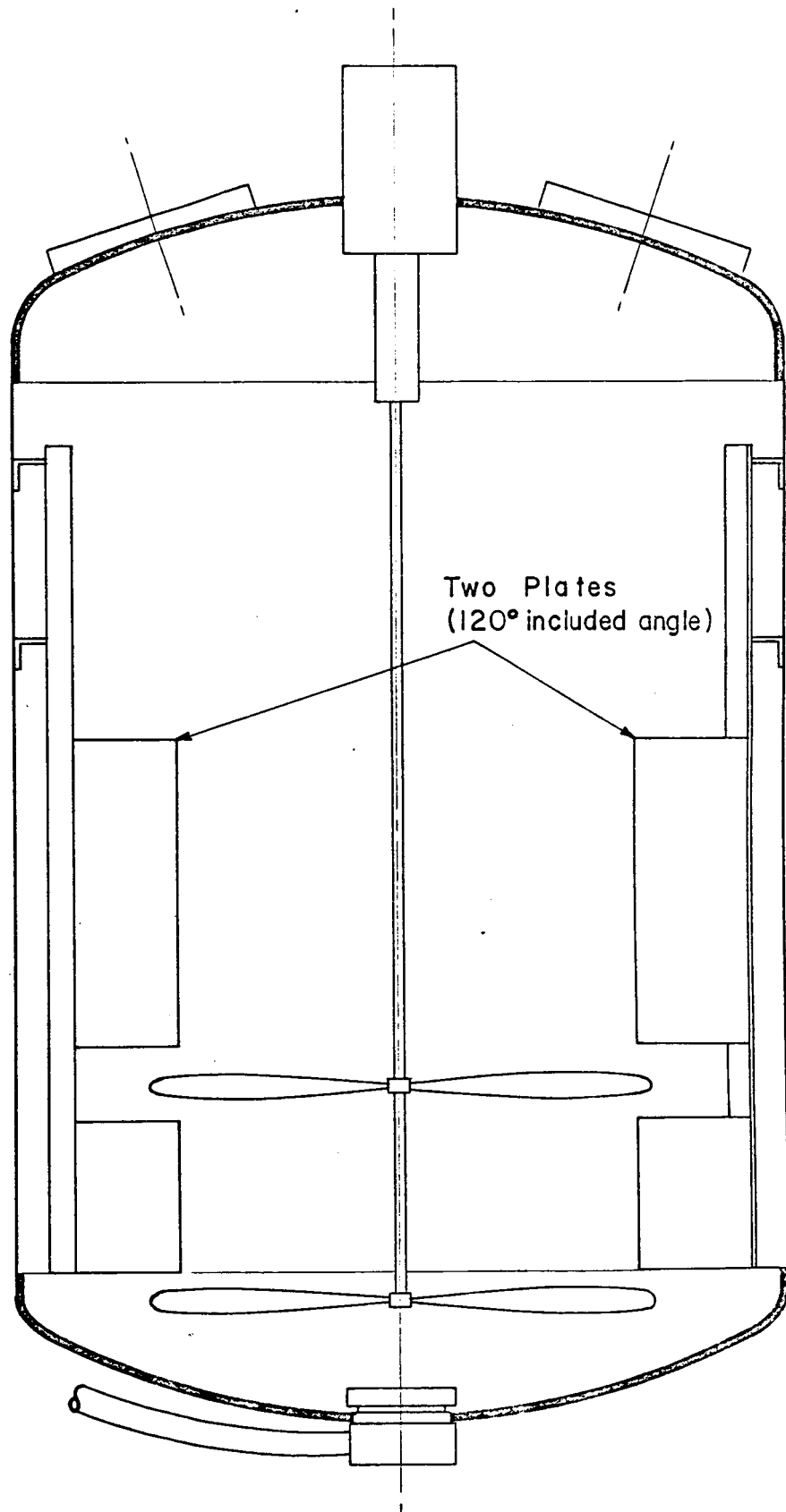
slush, it was found that the continuous stirring caused vortexing. Also, it was found that stirring had to be discontinued at the time the liquid level crossed the level sensors. If stirring was continued while the liquid crossed a level sensor, an inaccurate volume flow rate was obtained due to surface disturbances and vortexing.

To reduce vortexing, baffles were installed in the dewar at two of the 120° spaced internal mounting brackets. A pictorial drawing of the baffles is shown in figure 8. Level sensors five and one were used for outflow measurements. The use of two level sensors spaced as far apart as practical, reduced the percent inaccuracy in the flow rate due to surface disturbances and due to level sensor response. An additional stirring blade was also added at the 14 inch level in order to maintain homogeneity through better stirring action.

Some difficulty in maintaining constant pressure in the generator has been encountered since installation of the baffles and the additional stirring blade. The baffles cause more solid to circulate to the surface of the liquid. This combined with more stirring action in the ullage gas as the blades are exposed, causes the hydrogen gas in the ullage to be in equilibrium with the triple-point liquid and solid. Destroying the stratified layer of liquid and gas results in the requirement of more helium gas to maintain pressure. In future runs, increased helium supply pressure and flow will be required to maintain constant pressure in the generator.

2.2 Slush Hydrogen Particle Study

During the present reporting period, four hydrogen runs have been made with the Slush Hydrogen Particle Study Apparatus. Liquid-solid mixtures of hydrogen have been stored for periods of 24, 38, 51 and 59 hours while measurements of the solid fraction of the settled slush and the heat leak were made. As a result of the experience gained during these runs, several modifications to the apparatus have been made.



BAFFLE ARRANGEMENT FOR SLUSH GENERATOR

Figure 8

Solid Fraction of Settled Hydrogen Slush

The solid fraction of settled slush is defined as the solid fraction by mass of the material in the settled portion of the slush,

$$F_s = \frac{m_s}{m_l + m_s} \quad , \quad (1)$$

where m_l is the mass of the liquid filling the spaces between the solid particles and m_s is the mass of the solid. Any clear liquid above the liquid-slush interface is not included in m_l .

The solid fraction is determined by measuring the mass of the vapor pumped off from the production dewar together with the heat leak, as described by Daney and Mann [1966]. The expression used to calculate the settled solid fraction is

$$F_s = \frac{7.61 m_v - \frac{Q_{HL}}{L_f}}{m - m_v - m_{lc}} \quad . \quad (2)$$

Here m_v is the mass of gas pumped off during the freeze-thaw production process measured by a wet test meter connected to the vacuum pump exhaust. The initial mass, m , is determined from a liquid level measurement, the dewar having been volume calibrated. The mass of the clear liquid above the settled slush m_{lc} is determined from the difference between the measurements of the liquid level and the slush level. The remaining terms are Q_{HL} , the heat leak into the dewar; and L_f the latent heat of fusion. The constant 7.61 is the best experimental value for the ratio of the refrigeration produced during the freeze-thaw process, to the latent heat of fusion of parahydrogen.

Figure 9 shows the experimental values of the solid fraction of aged slush for two runs as a function of the aging time. After 59 hours, the

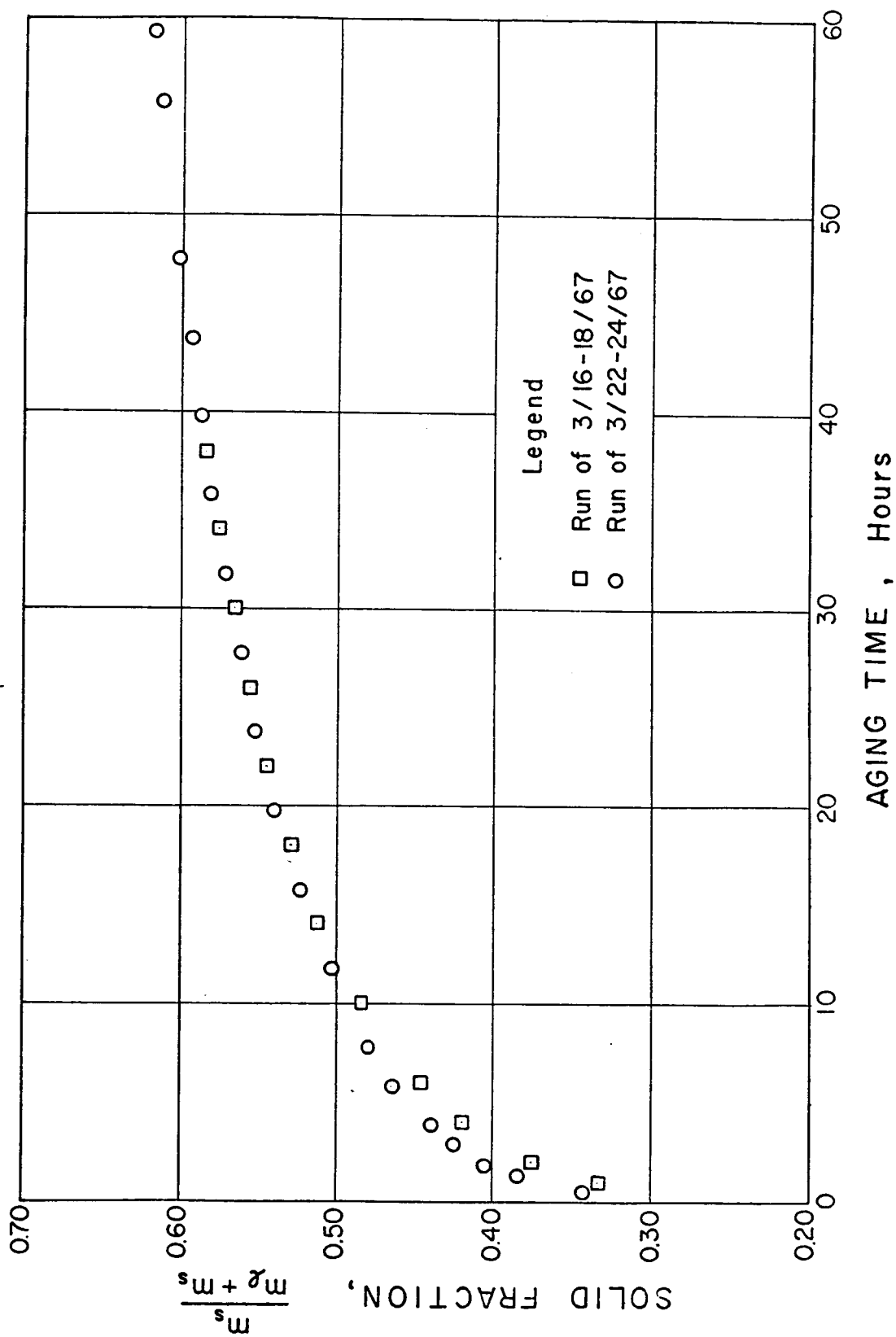


Figure 9. Solid Fraction of Aging Slush Hydrogen

greatest age obtained, the solid fraction reached a value of 0.62 and continued to increase at a slow rate.

When these aged batches of slush were stirred, they settled back to approximately their undisturbed density in less than a minute. No tendency for the solid particles to fuse together has been observed. In fact, several large multigrain pieces of solid, formed during two of the runs, were observed to disintegrate into their individual grains while aging.

Heat Leak

The heat leak into the stored slush is determined from the volume change due to melting as observed from the liquid level change. The average heat leak during the four runs was 1×10^{-4} watts with a standard deviation of 1×10^{-4} watts. The small change in the liquid level which occurs will make it difficult to determine the heat leak with much greater precision. However, this rate of heat leak amounts to a solid loss of only 0.3 percent per day. A liquid storage dewar with the same heat leak would lose 0.04 percent of its liquid per day. Thus the heat leak into the slush hydrogen is well below that that would be experienced in the field.

Conclusions

As a result of the work completed so far, some preliminary conclusions can be made. Solid mass fractions of at least 0.60 may be obtained by letting the slush age for two days or more. The aged slush will immediately settle back to a solid fraction within a few percent of the solid fraction observed before stirring. There is no tendency for individual solid particles to fuse together. Instead there is a tendency for larger, multigrain structures to break up as they age.

Finally it should be pointed out that aging is not a special treatment given to the mixture; it is a naturally occurring rearrangement of the particle structure that occurs inevitably.

2.3 NRA Slush Density Determination

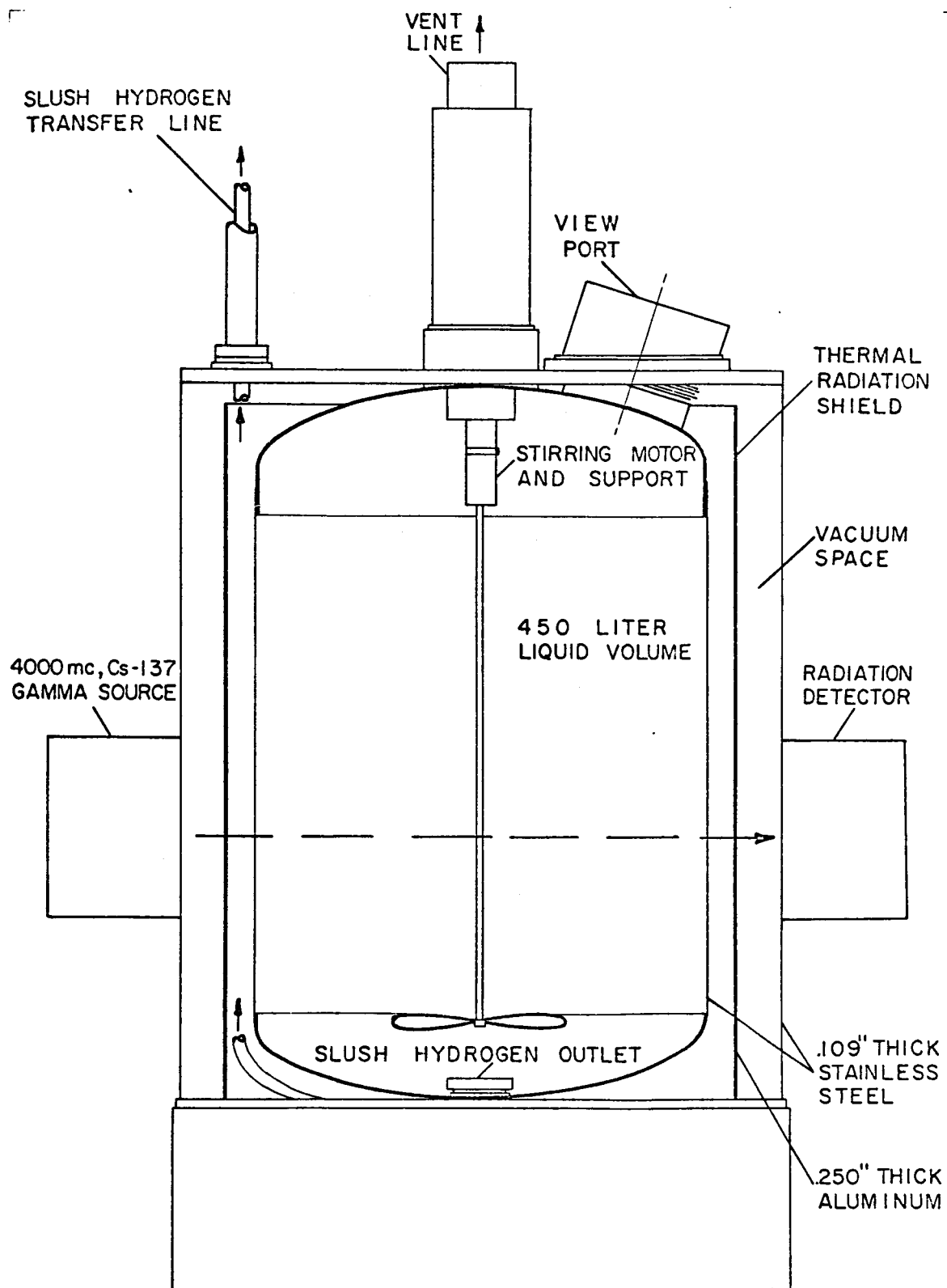
The Industrial Nucleonics Corporation AccuRay Density Measuring System has been installed on the 450 liter generator of the slush hydrogen flow loop. It consists of a 4 Curie Cesium 137 source, an ion chamber detector, an impedance matching unit, and an instrument console. Figures 10 and 11 show the position of the source and detector with respect to the generator. They are located on a generator diameter. A strip recorder and a digital volt meter have been connected to the instrument console to measure and record the signal from the densitometer.

While setting up the instrument, it was found that the ion chamber was damaged prior to arrival at this laboratory. Improper tightening of the set screws holding the ion chamber in place permitted it to fall out of position and crack the glass seal of the electrode. A new ion chamber has been loaned to us by Industrial Nucleonics Corporation.

They installed the new ion chamber and at the same time changed the hi-meg resistor from 10^9 ohms to 10^{10} ohms. This change increased both the signal and the time response by a factor of 10. The time response is now 1 sec.

Experimental Results

Figure 12 shows the densitometer output versus the density of liquid and liquid-solid mixtures of parahydrogen for a series of runs on five separate days. The densities in the liquid regions were determined by measuring the vapor pressure over the liquid in the generator while it was being vigorously stirred. The densities in the triple-point region were determined using the mass accounting method described by Daney and Mann [1966]. The densitometer output is measured by a digital volt meter connected to the recorder terminals of the instrument console.

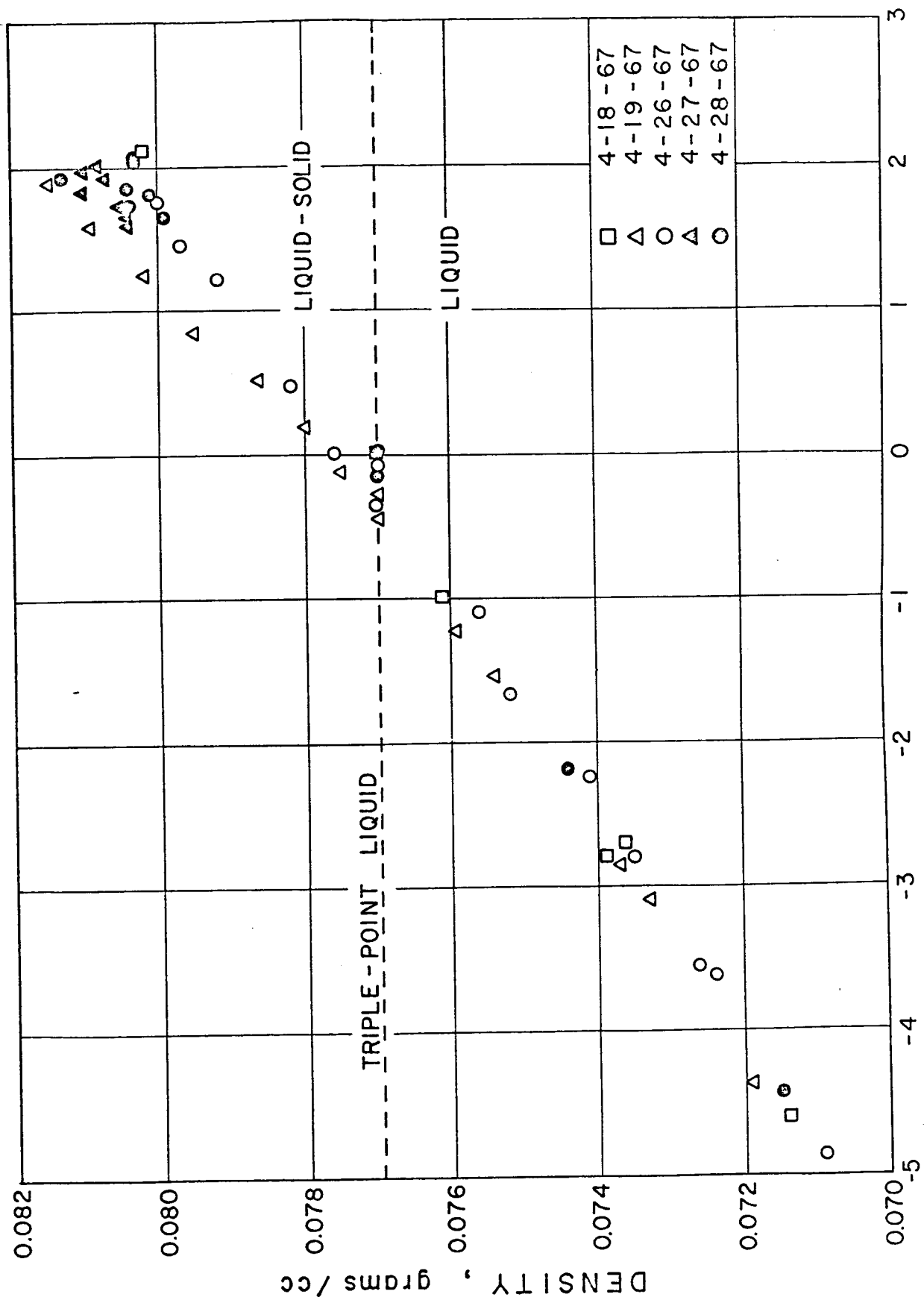


Existing Slush Generator

Figure 10



Figure 11.



DENSITOMETER OUTPUT, Millivolts

Figure 12

Discussion of Results

A detailed error analysis has not been completed, but some general comments can be made. In the liquid region the scatter of the points about a straight line is a few parts in 1000. The densitometer output at the triple-point liquid density varies by 0.4 millivolts. Since the triple-point liquid density is reproducible to about 1 point in 1000, we believe that a zero shift is occurring in the densitometer. The points in the liquid-solid region fall about a line with a different slope than those in the liquid region, and the scatter is much greater.

It is felt that the most likely reason for the scatter and difference in slope in the liquid-solid region is the uncertainty in our determination of the density in this region. In order to resolve this problem, we plan to install an electrical heater in the generator and obtain a calorimetric determination of the slush solid fraction.

High stirring rates in the generator can cause significant variations in the densitometer output when operating in the liquid-solid region. However, at moderate stirring rates, the output is essentially independent of the stirring rate.

A significant amount of noise in the output of the densitometer exists as shown in figure 13 which is a portion of the output as recorded on a strip recorder. The standard deviation of the signal to the digital volt meter (using a 1 sec. integration) is 0.06 millivolts. Placement of a 1/4 inch thick piece of lead in front of the beam with the generator empty yields approximately the same amount of noise. It is concluded then that the noise in the signal is not caused by density fluctuations in the hydrogen.

The experimental results presented are only preliminary, and more work must be done before any final evaluation of the densitometer can be made.

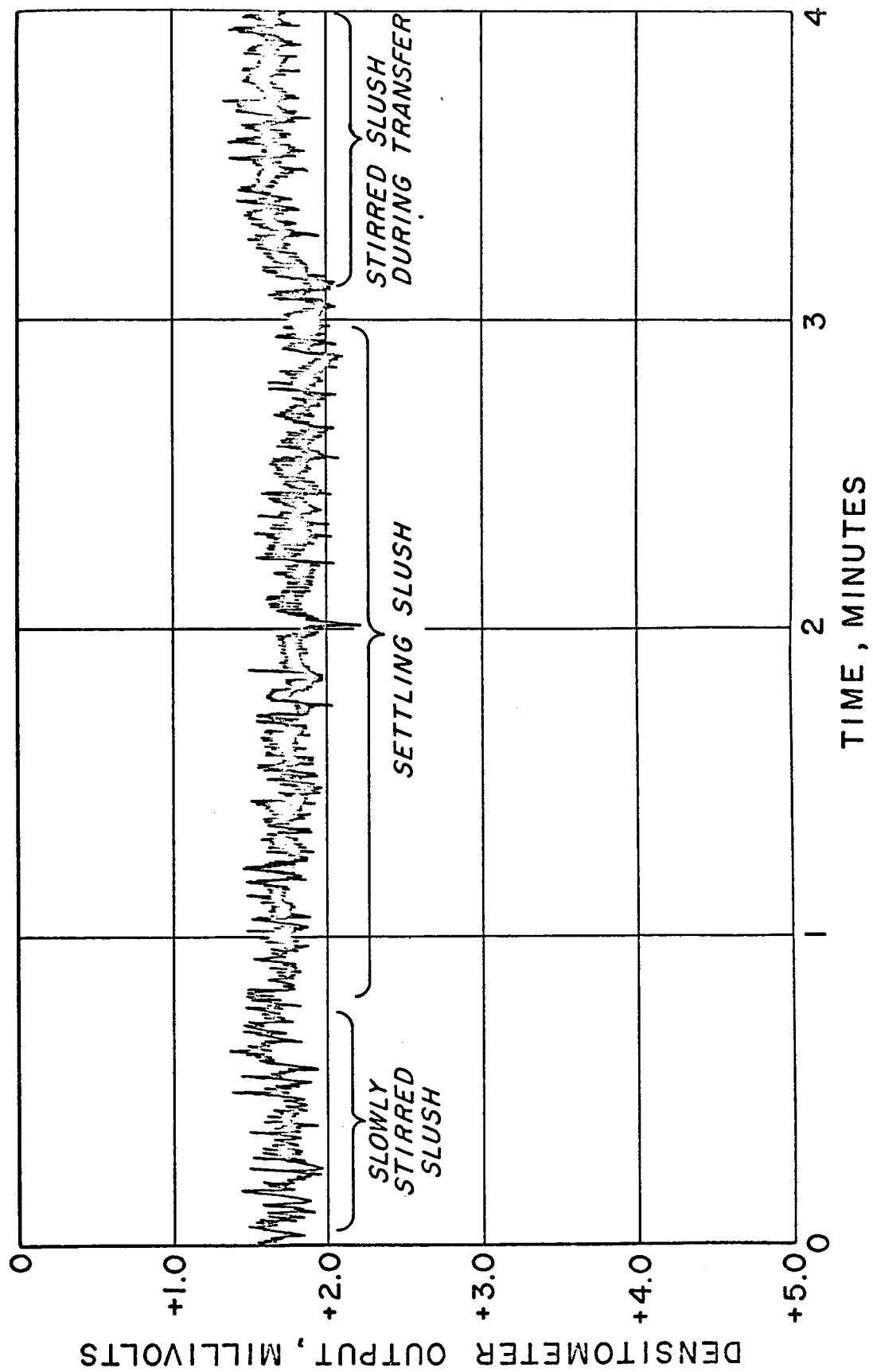


Figure 13

2.4 Slush Hydrogen Pump Test

Work has begun on the test set-up for studying the effects of pumping hydrogen slush with a PESCO LH_2 chilldown pump. The pump is of the centrifugal type with a nominal flow rate of 150 gallon/minute. It can be driven by either a 400 cycle/AC motor or by a helium gas driven turbine.

The pump will be installed in the 450 liter slush generator as described in previous discussions. A vacuum jacketed transfer line with a 2-inch inside diameter will permit the flow from the pump to be directed to either a 6000 liter dewar or returned to the slush generator. Two 1 1/2-inch globe valves will be used to direct the flow. This transfer line will be completely independent of the existing hydrogen slush transfer line, the exit and return to the dewar being made through a single window in the slush generator.

The design of the transfer line has been completed and the construction is in progress. The two vacuum insulated valves and a flexible section of transfer line, which will connect the rigid transfer line to the 6000 liter dewar, have been obtained.

3. FORTHCOMING REPORTING PERIOD

3.1 Slush Hydrogen Flow System

Additional flow data will be taken. Slush and triple-point liquid points will be obtained at higher flow rates and extremely low flow rates. All the flow data will be carefully evaluated with respect to flow correlations and the possibility of obtaining critical (low) velocities will be monitored.

3.2 Slush Hydrogen Particle Study

A run of 100 hours or more is planned for the immediate future. Measurement of the settled mass fraction and the dewar heat leak will again be made. The apparatus will then be modified to permit measurement of

of the angle of repose of the solid particles in both the fresh and the aged condition.

3.3 N. R. A. Slush Density Determination

In order to resolve the problems encountered in the liquid-solid region, we plan to install an electrical heater in the generator and make calorimetric measurements of the density in conjunction with measurements by the present method.

Investigation of the zero shift in the densitometer will also be made. Placement of a 1/4-inch thick piece of lead in the beam, with the dewar empty will give a constant value of attenuation in the operating region of the instrument. If a shift with time occurs, then it must be due to a zero shift in the instrument.

3.4 Slush Hydrogen Pump Test

The construction of the transfer line will continue. When the pump is received, the detailed design of its installation in the slush generator will be completed. Completion and installation of the transfer line, installation of the pump, and checking the system will occupy the next two months.